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(54) Process for producing a composite material consisting of gamma titanium aluminide as matrix with titanium diboride as perserdoid therein

Verfahren zur Herstellung einer Verbundwerkstoff, bestehend aus einem Matrix aus beta-Titanaluminid mit einer Dispersion von Titandiborid als Verstärkungsphase

Procédé pour la fabrication d'un matériau composite, constitué par une matrice de beta aluminiure de titane avec une dispersion de diborure de titane comme élément de renforcement

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a process for producing a TiB_2-dispersed TiAℓ-based composite material. More specifically, TiB_2 is uniformely dispersed in TiAℓ intermetallic compound-based matrix.

2. Description of the Related Art

The TiAℓ intermetallic compound is promising as a light-weight high temperature structural material since it has both metallic and ceramic properties, has a low density and has an excellent high temperature specific strength. The TiAℓ intermetallic compound is however limited in its applications since its hardness is low in comparison with normal metals and alloys.

To improve the hardness of the TiAℓ intermetallic compound, a TiAℓ-based composite material in which TiB_2 is dispersed was developed. For example, JP-A-03-193842, published in August, 1991, discloses a process for producing such a composite material, said process compressing mixing and melting powders of Aℓ matrix containing TiB_2 dispersed therein, Aℓ metal powders and Ti metal powders, followed by solidifying the same to form a TiAℓ intermetallic compound in which TiB_2 particles are dispersed.

As TiB_2 particles are dispersed in TiAℓ intermetallic compound, generally, the hardness of the TiAℓ intermetallic compound increases but the ductility thereof decreases. It is therefore necessary that TiB_2 particles are finely dispersed in the TiAℓ intermetallic compound. When the composite material is deformed, the matrix is deformed with cracks being formed. If the TiB_2 particles dispersed in the matrix are large, cracks are interrupted by the TiB_2 particles and the matrix cannot be deformed and is split or broken. In contrast, if the TiB_2 particles dispersed in the matrix are fine, cracks may develop through the gaps between the TiB_2 particles and the matrix can be deformed. Accordingly, it is considered that reduction of ductility of the matrix can be suppressed by finely dispersing TiB_2 particles in the matrix.

In the above mentioned process of producing a TiB_2-dispersed TiAℓ intermetallic compound-based composite material, however, it is difficult to finely disperse TiB_2 in a TiAℓ intermetallic compound since TiB_2 particles agglomerate with each other when the mixture of the TiB_2-dispersed Aℓ powders, Aℓ metallic powders and Ti metallic powders are melted.

The purpose of the present invention is to provide a process for producing a TiB_2-dispersed TiAℓ intermetallic compound-based composite material in which the dispersed TiB_2 is fine so that the reduction of the ductility of the material is suppressed while the hardness of the material is increased.

SUMMARY OF THE INVENTION

To attain the above and other objects of the present invention, there is provided a process for producing a TiB_2-dispersed TiAℓ-based composite material, comprising the steps of forming a molten mixture of a TiAℓ intermetallic compound source and a boride which is less stable than TiB_2, and cooling and solidifying said molten mixture to form a TiAℓ-based composite material in which TiB_2 is dispersed in an amount of 0.3 to 10% by volume of the composite material.

The TiAℓ intermetallic compound source may be a TiAℓ intermetallic compound itself, a mixture of Ti and Aℓ metal powders, or a mixture of the compound and the powder mixture. The composition of the source is preferably such that Aℓ is contained in an amount of 31 to 37% by weight of the total of Ti and Aℓ.

The boride should be less stable than TiB_2. Since TiB_2 is generally most stable among metal borides, most metal borides may be used in the present invention. Such borides include, for example, ZrB_2, NbB_2, TaB_2, MoB_2, CrB, WB, VB and HfB.

The particle size of the boride to be mixed is not particularly limited but preferably is less than 100 µm, more preferably 30 to 0.1 µm. If the particle size of the boride is larger than 30 µm, the time for decomposing the boride is elongated. If it is smaller than 0.1 µm, evaporation occurs during the melting step which reduces the yield.

The amount of the boride to be mixed is such that the obtained composite material will contain TiB_2 in an amount of 0.3 to 10% by volume, preferably 1 to 5% by volume, based on the composite material.

If the content of TiB_2 is less than 0.3% by volume, the hardness of the composite material is insufficient. If the content of TiB_2 is larger than 10% by volume, the ductility of the composite material is significantly lowered.

In the process for producing a TiB_2-dispersed TiAℓ intermetallic compound-based composite material of the present invention, a molten mixture of the TiAℓ intermetallic compound source and the boride is first formed. This molten mixture is typically formed by heating a powder mixture of the TiAℓ intermetallic compound source and the boride to a temper-

ature of about 1550 to 1750°C. If the temperature is lower than 1550°C, it is difficult to obtain a uniform dispersion of TiB_2. If the temperature is higher than 1750°C, the yield of Al is lowered. Alternatively, it is possible that the TiAℓ intermetallic compound source be first heated to form a molten TiAℓ intermetallic compound source, followed by adding the boron particles into the molten TiAℓ intermetallic compound source.

The molten mixture is then cooled to room temperature. During the cooling, the molten TiAℓ intermetallic compound source becomes a TiAℓ intermetallic compound and the added boron, which is less stable than TiB_2, reacts with Ti of the molten TiAℓ intermetallic compound source to crysptallize or deposite TiB_2 in the TiAℓ intermetallic compound matrix.

It is considered that the boride is dissolved and diffused in the molten Ti-Aℓ. Since TiB_2 is the most stable boride in the presence of Ti, boron (B), which became very fine by dissolution and diffusion of the boride, reacts with Ti to crystallize or deposite TiB_2. This reaction to form TiB_2 occurs uniformly in the molten mass so that fine TiB_2 is formed uniformly in the TiAℓ intermetallic compound.

The particle size of TiB_2 in the composite material may be made to be not larger than 10 μm, further not larger than 5 μm.

BRIEF DESCRIPTION OF DRAWINGS

Fig. 1 shows the microstructure of the TiB_2-dispersed TiAℓ-based composite material in Conventional Example 1 (× 100);
Fig. 2 shows the TiB_2 powders used to prepare the composite material of Fig. 1 (× 100); and
Fig. 3 shows the microstructure of the TiB_2-dispersed TiAℓ-based composite material in Example 6 (× 100).

Example 1

A mixture of a sponge Ti and an Aℓ ingot in a weight ratio of Aℓ/(Ti+Aℓ) of 0.34 was mixed with ZrB_2 powders with an average particle size of 3 μm in an amount of 3% by volume based on the volume of the total Ti-Aℓ. The thus obtained mixture was charged in a water-cooled copper crucible in an arc furnace and maintained in an argon atmosphere at a temperature between 1550°C and 1750°C for 10 minutes, followed by cooling in the crucible to produce a button ingot of a TiAℓ intermetallic compound matrix containing 2.52% by volume of TiB_2 dispersed therein.

Examples 2 to 8

The procedures of Example 1 were repeated, but the average particle size and amount of the boride to be mixed with the sponge Ti/Aℓ ingot mixture were varied as shown in Table 1. The button ingots of a TiAℓ intermetallic compound matrix containing TiB_2 particles dispersed therein in an amount as shown in Table 1 were produced.

Comparative Example 1

The procedures of Example 1 were repeated but the mixture of a sponge Ti and an Aℓ ingot in an Aℓ/(Ti+Aℓ) weight ratio of 0.34 was mixed with CrB powders with an average particle size of 30 μm in an amount of 0.2% by volume based on the volume of Ti-Aℓ, to thereby obtain a button ingot of a TiAℓ intermetallic compound matrix containing 0.15% by volume of TiB_2 particles dispersed therein.

Comparative Example 2

The procedures of Comparative Example 1 were repeated but the CrB powders mixed with the Ti-Aℓ was changed to 15% by volume.

Thus, a button ingot of a TiAℓ intermetallic compound matrix containing TiB_2 particles in an amount of 11.4% by volume was obtained.

Conventional Example 1

The procedures of Example 1 were repeated but the boride was changed to TiB_2 powders with an average particle size of 7 μm.

Thus, a button ingot of a TiAℓ intermetallic compound matrix containing 2.5% by volume of TiB_2 particles dispersed therein was obtained.

Conventional Example 2

A mixture of a sponge Ti and an Aℓ ingot in a weight ratio of Aℓ/(Aℓ+Ti) of 0.34 was mixed with B powders and, in accordance with the procedures of Example 1, a button ingot of a TiAℓ intermetallic compound matrix containing 2.4% by volume of TiB_2 particles dispersed therein was obtained.

Conventional Example 3

A sponge Ti and an Aℓ ingot were mixed in a weight ratio of Aℓ/(Ti+A) of 0.34 and charged in a water-cooled copper crucible in an arc furnace, in which the mixture was maintained in an argon atmosphere at a temperature of 1600 to 1700°C for 10 minutes and then cooled in the crucible to obtain a button ingot of a TiAℓ intermetallic compound.

Evaluations

Test pieces were cut from the button ingots of Examples 1 to 8, Comparative Examples 1 and 2, and Conventional Examples 1 to 3 and subjected to a Vickers hardness test and a bending test. The obtained hardness, elongation and bending strength of the test pieces are shown in Table 1.

TiB_2 was identified by X ray diffraction. The volume fraction of TiB_2 was determined by image analysis of micro structure of the composite.

Table 1

		Additive	Average particle size of additive (μm)	Amount of additive	Amount of TiB_2 in TiAℓ-based composite material (vol%)	Hardness (HV)	Elongation (%)	Bending strength (MPa)
Example	1	ZrB_2	3	3 vol%	2.52	355	0.90	880
	2	NbB_2	3	3 vol%	2.85	372	1.1	950
	3	TaB_2	3	3 vol%	2.85	350	1.05	965
	4	MoB	7	3 vol%	1.83	370	1.3	981
	5	CrB	30	0.5 vol%	0.38	307	1.4	927
	6	CrB	30	3 vol%	2.28	347	1.35	920
	7	CrB	30	10 vol%	7.6	395	0.95	988
	8	CrB	30	13 vol%	9.8	415	0.70	890
Comparative Example	1	CrB	30	0.2 vol%	0.15	280	1.40	930
	2	CrB	30	15 vol%	11.4	420	0.20	650
Conventional Example	1	TiB_2	7	3 vol%	2.5	351	0.55	779
	2	B	3	3 at%	2.4	355	0.45	750
	3	-	-	-	-	269	1.42	938

When the test pieces of Conventional Examples 1 and 2 in which TiB_2 particles were dispersed in a TiAℓ intermetallic compound matrix are compared with the test piece of Conventional Example 3 of a TiAℓ intermetallic compound, the test pieces of Conventional Examples 1 and 2 are superior in their hardness but inferior in their elongation and bending strength. It is considered that the above results are caused because the TiB_2 particles dispersed in the composite material are not fine. To confirm this, the microstructures of the test pieces of Conventional Example 1 and 2 were examined. Fig. 1 shows the microstructure of the test piece of Conventional Example 1 taken by microscope at a magnitude of 100. Fig. 2 shows the microstructure of the TiB_2 powders used for preparing the test piece of Conventional Example 1 at a magnitude of 100. From these microstructures, it becomes apparent that the particle size of the TiB_2 particles in the composite material in Conventional Example 1 increased from the 7 μm particle size of the original TiB_2 particles as mixed. A similar particle size increase was also found in the TiB_2 particles in Conventional Example 2. The reason for the increase of the TiB_2 particle size is thought because agglomeration of the TiB_2 particles.

The TiB_2-dispersed TiAℓ-based composite materials of Examples 1 to 8, i.e., produced in accordance with the process of the present invention, had improved elongation and bending strength in comparison with the test pieces of Conventional Examples 1 to 2, which are comparative to those of Conventional Example 3, and also had an excellent hardness. It is considered that the reason for the improved elongation and bending strength in Examples is because the particle size of the TiB_2 particles is finer. In the present invention, it is thought that the boride is dissolved and diffused in the molten Ti-Aℓ, the free boron released from the decomposed boride reacts with Ti in the molten Ti-Aℓ to form TiB_2, which is the most stable boride in the presence of Ti, and thus crystallizes or deposits fine TiB_2.

The microstructure of the test pieces of the Examples was examined. Fig. 3 shows the microstructure of the test piece of Example 6 taken by a microscope at a magnitude of 100. It is seen that the particle size of the TiB_2 particles ranges from the submicrons size to a few micro meters, that is, very fine. In other Examples, the particles sizes of the TiB_2 particles were found to be in the ranges from submicrons to a few micro meters.

It is thought that the elements other than B, such as Zr, Nb, Ta, Mo and Co, constituting the boride, are solid solved in the TiAℓ intermetallic compound and contribute to the improvement of the extension and hardness of the TiAℓ composite materials.

It is seen from Comparative Example 1 that if the content of the dispersed TiB_2 in the composite material is less than 0.3% by volume, an improved hardness i.e., a desired effect of dispersing the TiB_2 particles cannot be obtained. It is seen from Comparative Example 2 that if the content of the TiB_2 particles is more than 10% by volume, the hardness of the composite material is improved but the elongation and bending strength of the composite material are significantly decreased. The reason for the significant decrease of the elongation and bending strength of the composite material is thought to be because a portion of the boride particles cannot be dissolved and remain as large particles.

Accordingly, it is seen that the TiB_2 content of the TiB_2-dispersed TiAℓ-based composite material of the instant invention should be in a range of 0.3 to 10% by volume.

Claims

1. A process for producing a TiB_2-dispersed TiAℓ-based composite material, comprising the steps of:

    forming a molten mixture of a TiAℓ intermetallic compound source and a boride which is less stable than TiB_2, and
    cooling and solidifying said molten mixture to form a TiAℓ-based composite material in which TiB_2 is dispersed in an amount of 0.3 to 10% by volume of the composite material.

2. A process according to claim 1, wherein said boride is at least one selected from the group consisting of ZrB_2, NbB_2, TaB_2, MoB_2, CrB, WB, VB and HfB.

3. A process according to claim 2, wherein said boride has an average particle size of 100 to 0.1 μm.

4. A process according to claim 1, wherein said TiAℓ intermetallic compound source is a mixture of Ti and Aℓ metal particles, the Aℓ metal particles being in an amount of 31 to 37% by weight of the total of the Ti and Aℓ metal particles.

5. A process according to claim 1, wherein said TiAℓ intermetallic compound source includes a TiAℓ intermetallic compound.

6. A process according to claim 1, wherein said boride is added in such an amount that the obtained TiAℓ-based composite material contains 1 to 5% by volume of the dispersed TiB_2.

7. A process according to claim 1, wherein said mixture is heated up to a temperature of 1550°C to 1750°C.

8. A process according to claim 1, wherein said TiB_2 dispersed in said $TiA\ell$-based composite material has a particle size of less than 10 µm.

Patentansprüche

1. Verfahren zur Herstellung eines auf TiAl basierenden Verbundstoffmaterials mit darin dispergiertem TiB_2, welches die folgenden Schritte umfaßt:

   Ausbilden einer geschmolzenen Mischung eines Ausgangsstoffes einer intermetallischen TiAl-Verbindung und eines Borides, welches weniger stabil als TiB_2 ist, und

   Kühlen und Verfestigen der geschmolzenen Mischung, um ein auf TiAl basierendes Verbundstoffmaterial auszubilden, in welchem TiB_2 in einer Menge von 0,3 bis 10 Vol.-% des Verbundstoffmaterials dispergiert ist.

2. Verfahren gemäß Anspruch 1, wobei das Borid mindestens eines ist, ausgewählt aus der aus ZrB_2, NbB_2, TaB_2, MoB_2, CrB, WB, VB und HfB bestehenden Gruppe.

3. Verfahren gemäß Anspruch 2, wobei das Borid eine mittlere Teilchengröße von 100 bis 0,1 µm besitzt.

4. Verfahren gemäß Anspruch 1, wobei der Ausgangsstoff einer intermetallischen TiAl-Verbindung eine Mischung aus Ti- und Al-Metallteilchen ist, wobei die Al-Metallteilchen in einer Menge von 31 bis 37 Gew.-% der Gesamtheit der Ti- und Al-Metallteilchen vorhanden sind.

5. Verfahren gemäß Anspruch 1, wobei der Ausgangsstoff einer intermetallischen TiAl-Verbindung eine intermetallische TiAl-Verbindung enthält.

6. Verfahren gemäß Anspruch 1, wobei das Borid in einer solchen Menge zugegeben wird, daß das erhaltene auf TiAl basierende Verbundstoffmaterial 1 bis 5 Vol.-% des dispergierten TiB_2 enthält.

7. Verfahren gemäß Anspruch 1, wobei die Mischung auf eine Temperatur von 1550°C bis 1750°C erwärmt wird.

8. Verfahren gemäß Anspruch 1, wobei das in dem auf TiAl basierenden Verbundstoffmaterial dispergierte TiB_2 eine Teilchengröße von weniger als 10 µm besitzt.

Revendications

1. Un procédé de préparation de matériau composite à base de TiAl avec une dispersion de TiB_2 comprenant les étapes de :

   formation d'un mélange fondu d'une source de dérivé intermétallique TiAl et d'un borure qui est moins stable que TiB_2 et
   refroidissement et solidification du mélange fondu pour obtenir un matériau composite à base de TiAl dans lequel TiB_2 est dispersé à raison de 0,3 à 10% en volume dudit matériau composite.

2. Un procédé selon la revendication 1, dans lequel ledit borure est au moins un borure choisi dans le groupe consistant en ZrB_2, NbB_2, TaB_2, $M\sigma B_2$, CrB, WB, VB et HfB.

3. Un procédé selon la revendication 2, dans lequel ledit borure présente une dimension particulaire moyenne de 100 à 0,1 µm.

4. Un procédé selon la revendication 1, dans lequel ladite source de dérivé intermétallique TiAl est un mélange de particules métalliques de Ti et de Al, les particules de Al métallique étant présentes à raison de 31 à 37% du poids total des particules métalliques de Ti et de Al.

5. Un procédé selon la revendication 1, dans lequel ladite source de dérivé intermétallique TiAl comprend un dérivé intermétallique TiAl.

6. Un procédé selon la revendication 1, dans lequel ledit borure est ajouté en quantité telle que le matériau composite à base de TiAl obtenu contient de 1 à 5% en volume de TiB_2 dispersé.

7. Un procédé selon la revendication 1, dans lequel ledit mélange est chauffé à une température de 1550°C à 1750°C.

8. Un procédé selon la revendication 1, dans lequel le TiB_2 dispersé dans ledit matériau composite à base de TiAl présente une dimension particulaire inférieure à 10 µm.

Fig. 1

Fig. 2

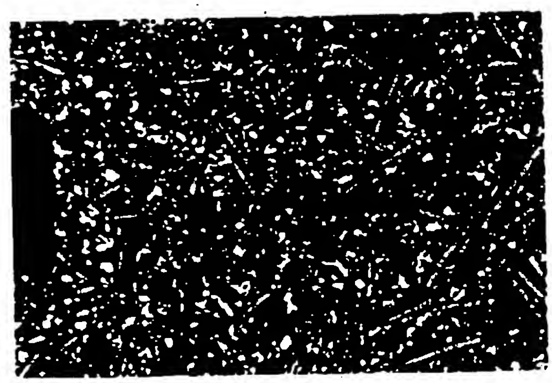

Fig. 3